ATTACHMENT N

START-3 QUALITY ASSURANCE SAMPLING PLAN

QUALITY ASSURANCE SAMPLING PLAN FOR NORPHLET CHEMICAL EMERGENCY RESPONSE 600 MACMILLAN ROAD NORPHLET, UNION COUNTY, ARKANSAS

Prepared For

U.S. Environmental Protection Agency Region 6 1445 Ross Ave. Dallas, Texas 75202

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April 2009

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1. INTRODUCTION

Weston Solutions, Inc. (WESTON®), the Superfund Technical Assessment and Response Team (START-3) contractor, has been tasked by the U.S. Environmental Protection Agency Region 6 (EPA Region 6) Prevention and Response Branch (PRB) under Contract Number EP-W-06-042, Technical Direction Document (TDD) Number TO-0001-09-04-03, to perform a Tier 3 emergency response at the Norphlet Chemical Company (NCC) located in Norphlet, Union County Arkansas just north of El Dorado. It was reported to the EPA that NCC is storing hydrofluoric acid (HF) in an unsafe manner that may lead to a HF release from the site. As a result, START-3 has been tasked to conduct air monitoring and sampling activities at the Norphlet site. START-3 has prepared this Quality Assurance Sampling Plan (QASP) to describe the technical scope of work to be completed at the site as part of the emergency response assessment.

1.1 PROJECT OBJECTIVES

START-3 is providing technical assistance to EPA Region 6 by collecting air samples for data necessary to support the EPA with public protection during the emergency removal operations and determination that the site no longer presents a threat to public health or welfare of the United States or the environment in accordance with 40 Code of Federal Regulations (CFR) 300.415.

The primary objective of the air sampling is to determine if levels are considered a health threat for the general public and the worker population in the area. The primary contaminants-of-concern (COCs) are hydrofluoric acid (HF) and tetrachloroethylene (TCE). The objectives of the sampling will be achieved by evaluating data obtained during the field activities through the collection of air samples from selected on-site locations.

An area determined by EPA On-scene Coordinator (OSC) Gary Moore will be initially monitored by START-3 using AreaRAE PGM-5010 multi-gas monitors and RKI Eagle HF monitors, if the monitors detect the COCs, than air sampling will be performed. The samples will be collected using SKC pumps and filters and sent to a commercial lab for analysis.

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1.2 PROJECT TEAM

The Project Team will consist of David Crow as the START-3 Emergency Response Manager; Robert Sherman as the START-3 Project Team Leader (PTL) and Field Safety Officer (FSO); and Tom Walzer as the Data Manager. Jeff Wright will serve as Alternate PTL and FSO as the project progresses. The PTL will be responsible for the technical quality of work performed in the field and will serve as the START-3 liaison to EPA Region 6 in the field during the site activities. The PTL, with the concurrence of the EPA OSC, will determine the locations for sample collection in the field, in collecting samples as necessary, in logging the activities at each sample location in the field logbook, and in verifying the sample documentation. The Data Manager, will be responsible for accurate chain-of-custody documentation for the samples collected during field activities. The Data Manager will oversee the packaging and shipping of samples to the designated laboratory, including the entry of daily sample collection into the Region Response Center – Electronic Data Management System (RRC-EDMS) Rapid Assessment Integrated Database (RAID) and removal modules software. The START-3 Field Safety Officer (FSO) will be responsible for providing overall site health and safety support.

1.3 QASP FORMAT

This QASP has been organized in a format that is intended to facilitate and effectively meet the objective of the emergency response. The QASP is organized as follows:

- Section 1 Introduction
- Section 2 Site Background
- Section 3 Sampling Approach and Procedures
- Section 4 Analytical Approach
- Section 5 Data Validation
- Section 6 Quality Assurance

All figures are provided as portable document format (pdf) files. Appendices are provided with the following information:

- Appendix A Site-Specific Data Quality Objectives (DQOs) Table
- Appendix B
 EPA ERT and WESTON Standard Operating Procedures (SOPs)
- Appendix C NIOSH Methods 1022 and 7903

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2. SITE BACKGROUND

Information regarding the site location, description, and history is included in the following subsections.

2.1 SITE LOCATION AND DESCRIPTION

Norphlet Chemical Company is located at 600 MacMillan (State Highway 335) at the former location of the MacMillan Oil Refinery Building in Norphlet, Union County, Arkansas, approximately 11 miles north of El Dorado. The chemical facility is approximately 92 acres and is located adjacent to a residential area. The Norphlet Elementary School borders the site to the west. The site is located within the boundaries of the town of Norphlet and is referenced in the El Dorado Quadrangle, U.S. Geological Survey (USGS) 7.5-minute quadrangle (Figure 2-1).

2.2 SITE HISTORY

NCC started physical operation in October 2007 as a chemical manufacturing facility specializing in producing refrigerant used in automobiles. The process of making the refrigerant involves hydrofluorination of TCE in the gas phase in the presence of fluorides (HF) as catalysts. Shortly after the company started, it was discovered they could not make the desired 1,1,1,2-tetrafluoroethane (R134a) at a profitable yield and ran out of funds in April 2008. The last employee of the facility left 11 October 2008.

NCC has five tanks on-site containing 31,800 gallons of HF in concentrations ranging from 4% to 98% that are stored in a manner that is potentially dangerous to life and health of the town of Norphlet. NCC is having difficulty removing the HF from the site and is involved in a legal battle with the HF manufacturer, Tulstar. The storage tanks are in dilapidated conditions and are located in close proximity to the entire Norphlet School District system. Norphlet Elementary school was closed on 17 April 2009 as a precaution.

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On 17 April 2009, START-3 mobilized to the site to begin perimeter air monitoring and collect data to determine if there is an air release that could pose an imminent and substantial threat to the community.

2.3 SITE CONCERNS

The primary site concern is the imminent threat of release of HF to the environment and the close proximity of the elementary school and surrounding community.

HF is one of the strongest inorganic acids. It is extremely corrosive and dangerous to handle. Exposure to HF can produce harmful health effects that may not be immediately apparent. HF is corrosive and readily destroys tissue. Exposure of the eyes to HF may result in blindness or permanent eye damage. HF readily penetrates human skin, allowing it to destroy soft tissues and decalcify bone. Inhaling HF vapors can seriously damage the lungs. Delayed reactions up to and including fatal pulmonary edema may not be apparent for hours after the initial exposure.

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3. SAMPLING APPROACH AND PROCEDURES

The specific field investigation activities that will be conducted during site sampling are presented in the following subsections. Sampling procedures and sample locations are also included.

3.1 OVERVIEW OF SAMPLING ACTIVITIES

The nature and extent of site-related contamination will be evaluated by air monitoring and collecting and analyzing air samples from selected locations determined by the EPA OSC with assistance from the START-3 PTL. The analytical results will be compared to the Occupational Safety and Health Administration (OSHA) Permissible Exposure Limit (PEL) of 3 parts per million (ppm) or 2.5 milligram per cubic meter (mg/m³) for HF and of 100 ppm or 537 mg/m³ for TCE.

3.1.1 Data Quality Objectives

The purpose of this project is to collect data that accurately represents the human health and environmental risks associated with this site. To accomplish this, a data quality objective (DQO) for determining the extent of contaminants in air has been established and is included in Appendix A. The DQOs presented were developed using the seven-step process set out in the EPA Guidance for Quality Assurance Project Plans: EPA QA/G-5. Air samples will be collected if air monitoring indicates a contaminant hit.

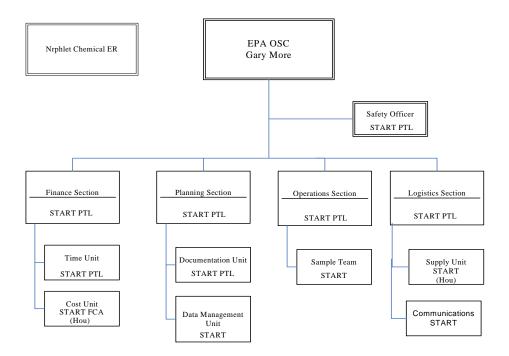
3.1.2 Field Activities Review Meeting

Prior to mobilizing to the site, the START-3 PTL will conduct a meeting with the entire field team to familiarize them with the project scope of work, discuss the planned field activities and roles and responsibilities, and review the project Health and Safety Plan (HASP) and other relevant START-3 and EPA operating procedures. The field team will also be briefed on the project budget and expense reporting responsibilities.

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3.1.3 Incident Command System and Health and Safety Implementation

START-3 will provide planning functions consistent with activities and responsibilities of the Incident Command System (ICS). At the beginning of each operational period, a daily operation meeting will be held in the command post to discuss objectives of the operation period, division assignments, field instrumentation calibration and use, and health and safety protocol. Every afternoon, a planning meeting will be conducted to develop a daily Incident Action Plan (IAP) for the next operation period. As part of ICS, local response officials, including the fire and police departments and the local hospital, will be notified to preplan for emergencies that may occur during the course of the emergency response. The ICS organization chart established for this site is as follows:



The emergency response assessment will be conducted in accordance with the site-specific HASP. START-3 will conduct all site activities in the personal protective equipment (PPE) as stated in the site HASP. The FSO will be responsible for implementation of the HASP during the emergency response. In accordance with the START-3 general health and safety operating

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procedures, the START-3 personnel will drive the route to the hospital specified in the HASP prior to initiating sampling activities.

3.1.4 Mobilization and Command Post Establishment

The START-3 field team will mobilize the equipment required for the emergency response assessment from the START-3 warehouse in Dallas, TX and Weston's Regional Equipment Store (RES) warehouse located in Houston, Texas, as necessary. If possible, START-3 will use government furnished property.

3.2 SAMPLING/MONITORING APPROACH

Air sampling will be conducted in general accordance with EPA ERT and WESTON Standard Operating Procedures (SOPs) (Appendix B).

3.2.1 Air Monitoring

START-3 performed continuous 24-hour perimeter air monitoring to determine whether site contaminants were migrating off-site to adjoining properties and potentially posing a threat to human health and the environment. START-3 continued to perform daily air monitoring during removal operations form 20 May 2009 to 19 June 2009. Air monitoring stations were positioned along the perimeter of the site work area as depicted in Attachment F. The location of each station was strategically selected each day by START-3 upon reviewing the local climatological data. These stations included an Area-RAE ambient air monitor with HF, volatile organic compounds (VOCs), chlorine (Cl2), lower explosive limit (LEL), and oxygen (O₂) sensors. The monitors were equipped with a radio transceiver that relayed data from the monitor's data logger to a central computer in the command post at 2-minute averaged intervals. These monitoring results were continuously observed by START-3 personnel. If elevated HF readings were detected, secondary confirmation monitoring was conducted with RKI ambient air monitors with HF sensors. Air monitoring results were compared to Occupational Safety and Health Administration (OSHA) Permissible Exposure Limit (PEL) of 3 parts per million (ppm) for HF.

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3.2.2 Air Sampling

Air samples will be collected by following NIOSH methods 1022 for TCE and 7903 for HF. HF will be collected using a SKC pump connected to a washed silica gel solid sorbent tube at a flow rate of 0.2 to 0.5 liter/minute for a total of 3 to 100 total liters collected (Tables 3-2 and 3-3). TCE will be collected using a SKC pump connected to a coconut shell charcoal solid sorbent tube at a flow rate of 0.01 to 0.2 liter/minute for a total of 1 liter to 30 liters. Field sampling pumps will have their flow rates determined by a primary calibration unit (BIOS International, DryCal Unit).

3.2.3 Investigation-Derived Waste (IDW)

It is not anticipated that IDW will be generated during the air sampling activities. If IDW is generated from used personal protective equipment (PPE), it will be placed in garbage bags and disposed by START-3 at a permitted disposal facility.

3.2.4 Sampling and Sample Handling Procedures

Samples will be collected using equipment and procedures appropriate to the matrix, parameters, and sampling objectives. The volumes of the samples collected must be sufficient to perform the analyses requested. Samples must be stored in the proper types of containers and preserved in a manner that is appropriate for the analyses to be performed.

All clean, decontaminated sampling equipment and sample containers will be maintained in a clean, segregated area. All samples will be collected with clean, decontaminated equipment. All samples for laboratory analysis will be collected in solid sorbent tubes and placed in individually designated Ziploc bags. Sampling personnel will change gloves between each sample collection/handling. All samples will be assembled and catalogued prior to shipping to the designated laboratory.

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3.2.5 Quality Assurance/Quality Control Samples

START-3 will collect field duplicate samples as needed during the response sampling activities. Quality assurance/quality control (QA/QC) samples will be collected according to the following:

- Blind field duplicate air samples will be collected during sampling activities at locations selected by the START-3 PTL. The data obtained from these samples will be used to assist in the quality assurance of the sampling procedures and laboratory analytical data by allowing an evaluation of reproducibility of results. Efforts will be made to collect duplicate samples in locations where there is visual evidence of contamination or where contamination is suspected. Blind field duplicate samples will be collected at the rate of one duplicate for every 10 samples collected.
- Field blanks will be unused sample media supplied by the laboratory. The field blank will be handled like an environmental sample and transported to the laboratory for analysis. Field blanks are used to assess the potential introduction of contaminants from ambient sources to the sample-collected media during sample collection. Field blanks shall be collected and submitted once per day for each day of sampling.
- Lot blanks are samples of the collection media, from the same manufacturer's lot as those being used for sample collection, submitted to the laboratory for analysis to detect potential contamination or issues with the sampling media. A minimum of two lot blanks will be submitted for each different lot of asbestos air filter cassettes that are used.

3.3 SAMPLE MANAGEMENT

Specific nomenclature that will be used by START-3 will provide a consistent means of facilitating the sampling and overall data management for the project as defined in the WESTON Standard Operating Procedures (SOPs) provided in Appendix B. Any deviations from the sample nomenclature proposed below must be approved by the START-3 PTL. The general nomenclature consists of the following components:

- Geographic location or on-site sample location
- QA/QC type (normal, duplicate, rinsate blank, etc.)
- Sequence (e.g., which sample it represents)

An example nomenclature for the air sampling at the Norphlet site is as follows:

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AB-MMDDYY-QQ

Where:

- AB designates a fixed sampling site
- MMDDYY Month Day Year that sample collection started
- QQ designates data type
 - \circ N = normal
 - \circ D = duplicate
 - \circ F = field blank

Sample locations will be identified in the field, as each location is sampled, independent of the physical location of the sample.

3.4 SAMPLE PRESERVATION, CONTAINERS, AND HOLD TIMES

Once collected, the air sample tubes will be stored in baggies. The samples will be sent to the designated laboratory by a common carrier.

Samples that have been analyzed will be disposed of by the designated laboratory in accordance with the laboratory SOPs.

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Table 3-1 Air Monitoring Equipment

Norphlet Chemical Norphlet, Union County, Arkansas

Air Monitoring Instruments	Air Monitoring Locations	Number Of Units	Rationale
AreaRAE – HF, Cl2, PID, %LEL	Perimeter and Hotzone	1 Upwind; 2 Downwind (minimum)	To monitor community and worker safety
RKI Eagle – HF	Perimeter and Hotzone	2	To monitor community and worker safety Confirmation AreaRAE readings
PACIII withHCl sensor (HF = 0.6 response)	Personal	6	To monitor worker safety
Zelwieger: HF Tape	TBD	2	To monitor worker safety

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Table 3-2 Requirements for Containers, Preservation Techniques, Sample Volumes, and Holding Times

Norphlet Chemical Norphlet, Union County, Arkansas

Name	Analytical Methods	Container	Preservation ^a	Minimum Sample Volume	Maximum Sample Volume	Maximum Holding Time
TCE-Air	NIOSH 1022	Coconut Solid Sorbent Tube in Ziploc bags	NA	1 Liter	30 Liters	Not Determined
HF - Air	NIOSH 7903	Washed Silica Gel Solid Sorbent Tube in Ziploc Bags	NA	3 Liters	100 Liters	At least 21 days

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Table 3-3 Sample Collection and Analysis Summary

Norphlet Chemical Norphlet, Union County, Arkansas

Sample Location	Sample Collection Method	No. of Samples	Rationale	Analytical Method
Perimeter and Work Zones	NIOSH	Maximum of 9 per day	To monitor community and worker safety	TCE: NIOSH 1022 HF: NIOSH 7903

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4. ANALYTICAL APPROACH

Air samples collected by START-3 during the sampling will by analyzed by the Test America - Phoenix using methods NIOSH 1022 and 7903.

The address for TestAmerica is:

TestAmerica –sample receiving c/o Stephanie Stimson 4625 E. Cotton Center Blvd., Suite 189 Phoenix, AZ 85040 866-775-5227

5. DATA VALIDATION

Data produced under this QASP will be evaluated to determine compliance with the stated collection methods, type, and number of samples collected, sample handling, and correct analytical procedures. Data review will be conducted in the laboratory prior to data release to evaluate the validity of the sample batch. Two data quality indicators, precision and accuracy, will be used to assess the batch. Data verification is the steps taken to determine whether the quality requirements specified in the DQOs of this QASP have been met. Data verification will be performed by START-3 for all commercial lab data. For field activities, it is necessary to determine whether the samples were collected using the sampling design specified in Subsection 3.2, whether the samples were collected according to a specific method or SOP as specified in Subsection 3.3, whether the proper amount of QC samples were taken to satisfy the QC requirements specified in Subsection 3.2.4, and whether the collected samples have been recorded and handled properly as in Section 6. For analytical activities, each sample should be verified to ensure that the procedures used to generate the data (as specified in Section 4) were performed as specified. The proper amount of QC checks (as specified in Subsection 3.4) that were prepared and analyzed during the actual analysis provide an indication of the quality of the data. Instrument calibrations (as specified in Subsection 3.4) are evaluated to determine whether the correct number of calibration standards were used and the range of the analysis and whether standards were analyzed in an appropriate sequence specific to the methods used and were performed prior to the analysis of samples, blanks, and QC samples in an appropriate time frame.

6. QUALITY ASSURANCE

Quality assurance will be conducted in accordance with the WESTON Corporate Quality Management Manual, dated March 2004; the WESTON START-3 Quality Management Plan, dated May 2007; and EPA Quality Assurance/Quality Control Guidance for Sampling Activities, dated April 1990. Following receipt of the TDD from EPA, a Quality Control (QC) officer will be assigned and will monitor work conducted throughout the entire project including reviewing interim report deliverables and field audits. The START-3 PTL will be responsible for QA/QC of the field investigation activities. The designated laboratory utilized during the investigation will be responsible for QA/QC related to the analytical work. START-3 will also collect samples to verify that laboratory QA/QC is consistent with the required standards and to validate the laboratory data received.

6.1 SAMPLE CUSTODY PROCEDURES

Because of the evidentiary nature of sample collection, the possession of samples must be traceable from the time the samples are collected until they are introduced as evidence in legal proceedings. After sample collection and identification, samples will be maintained under COC procedures. If the sample collected is to be split (laboratory QC), the sample will be allocated into similar sample containers. Sample labels completed with the same information as that on the original sample container will be attached to each of the split samples. All personnel required to package and ship coolers containing potentially hazardous material will be trained accordingly.

START-3 personnel will prepare and complete COC forms using the SCRIBE environmental sampling data management system for all samples sent to a START-3 designated off-site laboratory. The COC procedures are documented and will be made available to all personnel involved with the sampling. A typical COC record will be completed each time a sample or group of samples is prepared for shipment to the laboratory. The record will repeat the information on each sample label and will serve as documentation of handling during shipment. A copy of this record will remain with the shipped samples at all times, and another copy will be

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retained by the member of the sampling team who originally relinquished the samples. At the completion of the project, the Data Manager will export the SCRIBE COC documentation to the Analytical Service Tracking System (ANSETS) database.

Samples relinquished to the participating laboratories will be subject to the following procedures for transfer of custody and shipment:

- Samples will be accompanied by the COC record. When transferring possession of samples, the individuals relinquishing and receiving the samples will sign, date, and note the time of the sample transfer on the record. The custody record documents the transfer of sample custody from the sampler to another person or to the laboratory.
- Samples will be properly packed for shipment and dispatched to the appropriate laboratory for analysis with separate, signed custody records enclosed in each sample box or cooler. Sample shipping containers will be custody-sealed for shipment to the laboratory. The preferred procedure includes use of a custody seal wrapped across filament tape that is wrapped around the package at least twice. The custody seal will then be sealed to ensure that the only access to the package is by cutting the filament tape or breaking the seal to unwrap the tape.
- If sent by common carrier, a bill of lading or air bill will be used. Bill of lading and air bill receipts will be retained in the project file as part of the permanent documentation of sample shipping and transfer.

START-3 SOPs 1101.01 and 1102.01 describe these procedures in more detail.

6.2 PROJECT DOCUMENTATION

All documents will be completed legibly and in ink and by entry into field logbooks, Response Manager, or SCRIBE. Response Manager is the Enterprise Data Collection System designed to provide near real-time access to non-analytical data normally collected in logbooks. Response Manager provides a standard data collection interface for modules of data normally collected by START-3 field personnel while on-site. These modules fall into two basic categories for Response and Removal. The modules include Emergency Response, Reconnaissance, Facility Assessment, Shipping, Containers, Materials, Calls, HHW, and General/Site Specific data. The system provides users with a standard template for laptop/desktop/tablet PCs that will synchronize to the secure web interface using merge replication technology to provide access to

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field collected data via on the RRC-EDMS EPA Web Hub. Response Manager also includes a PDA application that provides some of the standard data entry templates from Response Manager to users for field data entry. Response Manager also includes an integrated GPS unit with the secure PDA application, and the coordinates collected in Response Manager are automatically mapped on the RRC-EDMS interactive mapping site. GIS personnel can then access this data to provide comprehensive site maps for decision-making support.

Response Manager also includes an Analytical Module that is designed to give Scribe users the ability to synchronize the SCRIBE field data to the RRC-EDMS Web Hub. This allows analytical data managers and data validators access to data to perform reviews from anywhere with an Internet connection. The Analytical Module is designed to take the analytical data entered into EPA SCRIBE software and make it available for multiple users to access on one site. START-3 personnel will utilize SCRIBE for all data entry on-site and will upload to the Response Manager Analytical module.

6.2.1 Field Documentation

The following field documentation will be maintained as described below.

Field Logbook

The field logbook is a descriptive notebook detailing site activities and observations so that an accurate, factual account of field procedures may be reconstructed. All entries will be signed by the individuals making them. Entries should include, at a minimum, the following:

- Site name and project number.
- Names of personnel on-site.
- Dates and times of all entries.
- Description of all site activities, including site entry and exit times.
- Noteworthy events and discussions.
- Weather conditions.
- Site observations.
- Identification and description of samples and locations.
- Subcontractor information and names of on-site personnel.
- Dates and times of sample collections and chain-of-custody information.
- Records of photographs.

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- Site sketches.
- Calibration results.

Sample Labels

Sample labels will be securely affixed to the sample container. The labels will clearly identify the particular sample and include the following information:

- Site name and project number.
- Date and time the sample was collected.
- Sample preservation method.
- Analysis requested.
- Sampling location.

Chain-of-Custody Record

A chain-of-custody will be maintained from the time of sample collection until final deposition. Every transfer of custody will be noted and signed for and a copy of the record will be kept by each individual who has signed it. The chain-of-custody is discussed in Subsection 6.1 Sample Custody Procedures.

Custody Seal

Custody seals demonstrate that a sample container has not been tampered with or opened. The individual who has custody of the samples will sign and date the seal and affix it to the container in such a manner that it cannot be opened without breaking the seal.

Photographic Documentation

START-3 will take photographs to document site conditions and activities as site work progresses. Initial conditions should be well documented by photographing features that define the site-related contamination or special working conditions. Representative photographs should be taken of each type of site activity. The photographs should show typical operations and operating conditions as well as special situations and conditions that may arise during site activities. Site final conditions should also be documented as a record of how the site appeared at completion of the work.

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All photographs should be taken with either a film camera or digital camera capable of recording the date on the image. Each photograph will be recorded in the logbook and within Response Manager with the location of the photographer, direction the photograph was taken, the subject of the photograph, and its significance (i.e., why the picture was taken). Where appropriate, the photograph location, direction, and subject will also be shown on a site sketch and recorded within Response Manager.

6.2.2 Report Preparation

At the completion of the project, START-3 will review and validate the laboratory data and prepare a draft report of field activities and analytical results for EPA OSC review. Draft deliverable documents will be uploaded to the EPA TeamLink website for EPA OSC review and comment.

6.2.3 Response Manager

START-3 will use the Response Manager module located on the EPA Web Hub, https://solutions.westonproject.net/epawebhub/, to collect and organize the data collected from project activities. The information to be included encompasses some or all of the following depending on the specific project needs:

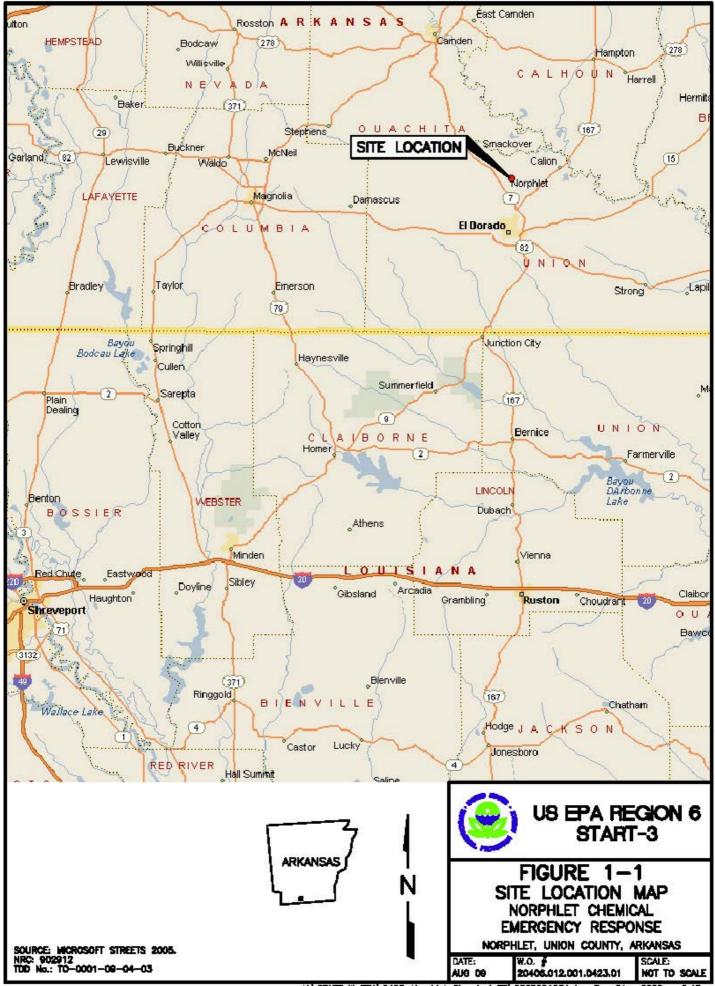
- General Module Site specific data including location and type of site. It also includes an area for all key site locations including geo-spatial data associated with the key site locations.
- Emergency Response Module includes the following sub-modules: Basic Info, HAZMAT, Release, Timeline log, Incident Zones, Photos, Sensitive Receptors, Evacuations, Source, Cause, and Weather.
- Reconnaissance Module provides standard templates with the flexibility of adding any
 additional question's of values to the drop down lists for targeted reconnaissance efforts.
 Typically the data in this module is associated with ESF-10 deployments and the cleanup of orphaned containers and hazardous debris, but the module can be utilized for any
 an all reconnaissance activities.

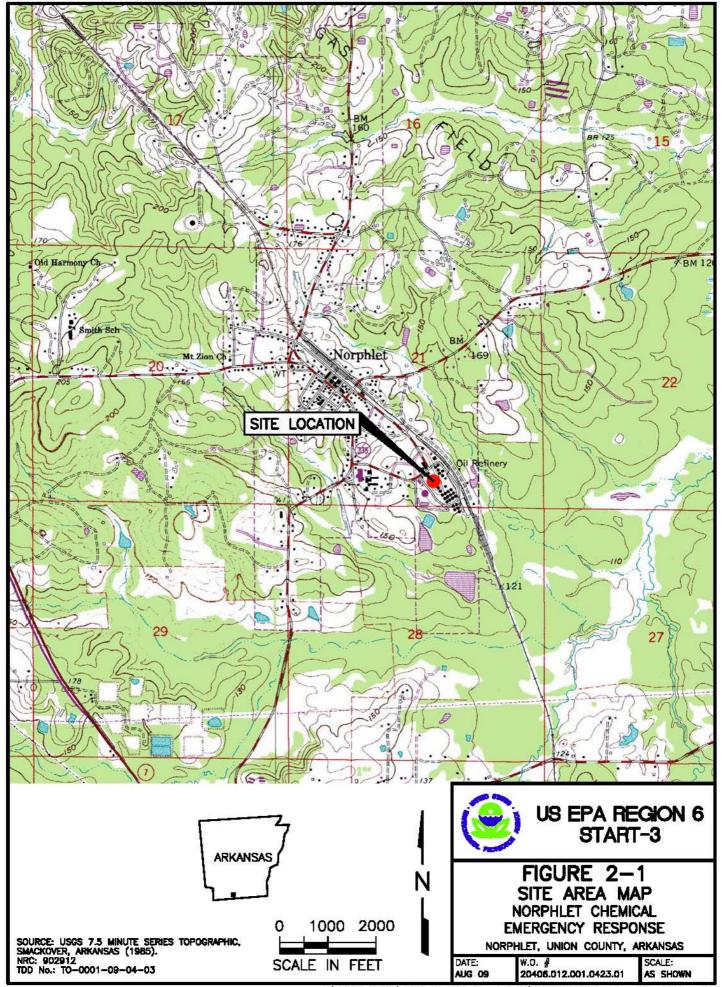
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- Facility Assessment Module provides standard templates with the flexibility of adding any additional question's of values to the drop down lists for assessments of structures. Typically utilized for EPA regulated program facilities during an ESF-10 deployment of resources. This module can be utilized to track the assessment of any facilities including multiple assessments of the fixed facilities.
- Shipping Module provides standard templates for creating a cradle to grave record of all waste shipments from the site until they are recycled or destroyed. This includes the ability to capture manifest and manifest line items, and upload photos/original documents to support the records.
- Container Module provides standard templates for cataloguing containers including HAZCAT and Layer information in each container. The module also allows for the tracking of which containers are bulked.
- Properties Module provides standard templates with the flexibility of adding any
 additional question's of values to the drop down lists for collection of property data
 including access agreements and assessments of the property and current status of
 property with regards to the site removal action.
- Materials Module provides standard templates for tracking materials that are brought on-site or that are removed from the site.
- Daily Reports provides standard templates for tracking daily site activities, daily site personnel, and daily site notes for reporting back to the EPA OSC in a POLREP or SITREP.
- HHW Module provides standard templates with the flexibility of adding any additional questions of values to the drop down lists for tracking the amount of HHW collected at individual collection stations by HHW type.
- Data Files data files can be uploaded in the photo module section and be associated
 with individual records or with the site in general. The meta data associated with that
 data file can be filled in using the photo log fields.

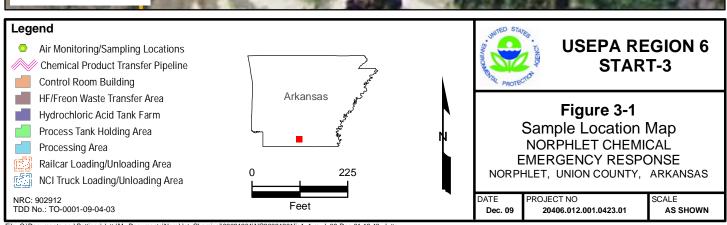
The data stored in the Response Manager database can be viewed and edited by any individual with access rights to those functions. At anytime deemed necessary, POLREP and/or SITREP's can be generated by exporting the data out of Response Manager into Microsoft Excel/Word. The database is stored on a secure server and backed up regularly.

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APPENDIX A

Site-Specific Data Quality Objectives Tables

DATA QUALITY OBJECTIVE NO. 1 MEDIA OF CONCERN: AIR

STEP 1. STATE THE PROBLEM

Hydrofluoric acid (HF) and Tetrachloroethylene (TCE) present in the atmosphere.

STEP 2. IDENTIFY THE DECISION

Is the concentration of contaminants of concern in air, represented by air monitoring or sampling, are above specified Health and Safety levels?

IDENTIFY THE ALTERNATIVE ACTIONS THAT MAY BE TAKEN BASED ON THE DECISIONS.

- If the contaminant is detected with air monitoring equipment and exceeds the assigned action levels in air then air samples will be collected for the COC. If the analytical result exceeds the specified action level then the air will be considered contaminated and appropriate engineering controls will be applied.
- If the contaminants do not exceed the specified action levels in air, the air represented by that data will not require additional attention.

STEP 3. IDENTIFY INPUTS TO THE DECISION

IDENTIFY THE INFORMATIONAL INPUTS NEEDED TO RESOLVE A DECISION.
IDENTIFY THE SOURCES FOR EACH

Contaminant concentrations in air samples.

- IDENTIFY THE SOURCES FOR EACH INFORMATIONAL INPUT AND LIST THE INPUTS THAT ARE OBTAINED THROUGH ENVIRONMENTAL MEASUREMENTS.
- Air sample locations
- Analytical results from HF and ECE analyses.

BASIS FOR THE CONTAMINANT SPECIFIC ACTION LEVELS.

The action level is dependent on the results and whether it is determined that a release is occurring.

IDENTIFY POTENTIAL SAMPLING TECHNIQUES AND APPROPRIATE ANALYTICAL METHODS.

Table 3-1 specifies sampling technique.

- NIOSH 7903 for HF
- NIOSH 1022 for TCE

DATA QUALITY OBJECTIVE NO. 1 MEDIA OF CONCERN: AIR (Cont'd)

STEP 4. DEFINE THE BOUNDARIES OF	STEP 4. DEFINE THE BOUNDARIES OF THE STUDY			
DEFINE THE DOMAIN OR GEOGRAPHIC AREA WITHIN WHICH ALL DECISIONS MUST APPLY.	Air monitoring and air sampling stations determined by START-3 PTL and EPA OSC.			
SPECIFY THE CHARACTERISTICS THAT DEFINE THE POPULATION OF INTEREST.	Contaminant concentrations in air at the sample locations.			
DEFINE THE SCALE OF DECISION MAKING.	The scale of decision will be for the air represented by each sample collected.			
DETERMINE THE TIME FRAME TO WHICH THE DATA APPLY.	The data will apply until the area represented by the sample receives appropriate response actions.			
DETERMINE WHEN TO COLLECT DATA.	Samples will be collected following air monitoring if detections are noted.			
IDENTIFY PRACTICAL CONSTRAINTS ON DATA COLLECTION.	Inclement weather.			
STEP 5. DEVELOP A DECISION RULE				
SPECIFY THE PARAMETER THAT CHARACTERIZES THE POPULATION OF INTEREST.	The sample concentrations at each sample location will be compared to the site-specific action levels.			
SPECIFY THE ACTION LEVEL FOR THE DECISION.	3 ppm or 2.5 mg/m ³ for HF and 100 ppm or 537 mg/m ³ for TCE.			
DEVELOP A DECISION RULE.	If any result in an air sample is above the contaminant specific screening level, then area represented by that sample will require additional response actions, otherwise the area will continue to be monitored until the emergency response removal is completed.			

DATA QUALITY OBJECTIVE NO. 1 HURRICANE GUSTAV

MED	IA OF	CONCERN:	AIR (Cont'd)

STEP 6. SPECIFY LIMITS ON DECISION	ERRORS
DETERMINE THE POSSIBLE RANGE OF THE PARAMETER OF INTEREST.	Contaminant concentrations may range from non-detect to above the screening values for soil.
DEFINE BOTH TYPES OF DECISION ERRORS AND IDENTIFY THE POTENTIAL CONSEQUENCES OF EACH.	Type I Error: Deciding that the specified area represented by the air sample does not exceed the specified action level when, in truth, the soil concentration of the contaminant exceeds its action level. The consequence of this decision error is that contaminants will continue to be present in the atmosphere, possibly endangering human health and the environment. This decision error is more severe.
DEFINE BOTH TYPES OF DECISION ERRORS AND IDENTIFY THE POTENTIAL CONSEQUENCES OF EACH.	Type II Error: Deciding that the specified area represented by the air sample does exceed the specified action level when, in truth, it does not. The consequences of this decision error are that emergency removal actions will be stopped and altered and unnecessary costs will be incurred.
ESTABLISH THE TRUE STATE OF NATURE FOR EACH DECISION RULE.	The true state of nature when the air in the area is decided to be below the action levels when in fact, it is not below the specified action levels, is that further characterization and removal actions may be necessary. The true state of nature when the air in the area is decided to be above the specified action levels when in fact, it is not above the specified action levels, is that the removal action method does not need to be altered.
DEFINE THE TRUE STATE OF NATURE FOR THE MORE SEVERE DECISION ERROR AS THE BASELINE CONDITION OR THE NULL HYPOTHESIS (H _o) AND DEFINE THE TRUE STATE FOR THE LESS SEVERE DECISION ERROR AS THE ALTERNATIVE HYPOTHESIS (H _a).	H _o : The air represented by the air sample is above the specified action level. H _a : The air represented by the air sample is below the specified action level.

DATA QUALITY OBJECTIVE NO. 1 HURRICANE GUSTAV MEDIA OF CONCERN: AIR (Cont'd)

STEP 6. SPECIFY LIMITS ON DECISION	ERRORS (Cont'd)
ASSIGN THE TERMS "FALSE POSITIVE" AND "FALSE NEGATIVE" TO THE PROPER DECISION ERRORS.	 False Positive Error = Type I False Negative Error = Type II
ASSIGN PROBABILITY VALUES TO POINTS ABOVE AND BELOW THE ACTION LEVEL THAT REFLECT THE ACCEPTABLE PROBABILITY FOR THE OCCURRENCES OF DECISION ERRORS.	The assignment of probability values is not applicable to this DQO because these samples are being collected for baseline and screening purposes.
STEP 7. OPTIMIZE THE DESIGN	
REVIEW THE DQOs.	Due to insufficient historical data, determination of the standard deviation was not possible. Therefore, sample size calculation using the traditional statistical formula may not be the optimal design. In order to select the optimal sampling program that satisfies the DQOs and is the most resource effective, other elements were considered.

DEVELOP GENERAL SAMPLING AND ANALYSIS DESIGN.

Air monitoring will be collected using AreaRAE PGM-5010 multi-gas monitors and RKI Eagle HF monitors. If the monitors detect the COCs, then air sampling will be performed. The air samples will be collected using SKC pumps at locations determined by the START-3 PTL and EPA OSC. The samples will be submitted to TestAmerica in Phoenix, AZ for analytical analysis.

APPENDIX B

EPA ERT and WESTON Standard Operating Procedures

SOP	0110.04					
GROUP	Database Management	Database Management System				
SUB-GROUP	Data Collection and Ad	equisition				
TITLE	On-Site Sample Nome	nclature - On-Site	e Sampling Activities			
DATE	3/19/2004	FILE	0110-04.DOC	PAGE	l of l	

The following Standard Operating Procedure (SOP) presents the remediation sample nomenclature for analytical samples. The sample nomenclature is based upon specific code requirements for compatibility with the WESTON On-Line system

PROCEDURE

Sampling Stations.

Station Type	Template
Soil Stockpile	SS##
Monitoring Well	MW##
Surface Water Pond	POND##
Air Sampler	AIR##

Sample Nomenclature.

Sample Type	Template	Example
Soil Composite Sample	Stockpile - Type - QC - Sequence	SS01-CO-N-1
Surface Water Sample	Surface Water Pond-Type-QC-Sequence	POND03-CO-N-1
Groundwater Sample	Monitoring Well-Type-QC-Sequence	MW12-CO-N-1
Ambient Air Sample	Air Sampler-Sample Type-QC Type-Sequence	AIR01-TI-N-1

Note: Sequence is a numeric counter to make Sample ID unique if more than one sample is collected.

Sample Types.

Sample Type Description	Code	
Composite	CO	
Grab	G	1
Product - DNAPL	PD	
Product - LNAPL	PL	1
Split	SP	1
Time Integrated	TI	

QA/QC Types.

QA/QC Type Description	Code
Normal	N
Duplicate	D
Field Blank	FB
Rinse Blank	RB
Trip Blank	TB

SOP	1101.01				
GROUP	Sampling Handling	Carried St.			
SUB-GROUP	Sample Custody				
TITLE	Sample Custody in the	Field			
DATE	11/12/2003	FILE	1101-01.DOC	PAGE	1 of 4

The following Standard Operating Procedure (SOP) presents procedures for maintaining sample chain of custody (COC) during activities where samples are collected.

PROCEDURE

Sample custody is defined as being under a person's custody if any of the following conditions exist:

- · it is in their possession.
- · it is in their view, after being in their possession,
- · it was in their possession and they locked it up, or
- it is in a designated secure area.

A designated field sampler will be personally responsible for the care and custody of collected samples until they are transferred to another person or properly dispatched to the laboratory. To the extent practicable, as few people as possible will handle the samples.

Sample tags or labels will be completed and applied to the container of each sample. When the tags or labels are being completed, waterproof ink will be used. If waterproof ink is not used, the tags or labels will be covered by transparent waterproof tape. Sample containers may also be placed in Ziploc-type storage bags to help keep them clean in the cooler. Information typically included on the sample tags or labels will include the following:

- Project Code
- Station Number and Location
- Sample Identification Number
- · Date and Time of Sample Collection
- Type of Laboratory Analysis Required
- Preservation Required, if applicable
- Collector's Signature
- Priority (optional)
- Other Remarks

Additional information may include:

- · Anticipated Range of Results (Low, Medium, or High)
- Sample Analysis Priority

SOP	1101.01					
GROUP	Sampling Handling					
SUB-GROUP	Sample Custody					
TITLE	Sample Custody in the	Field				
DATE	11/12/2003	FILE	1101-01.DOC	PAGE	2 of 4	

A COC form will be completed each time a sample or group of samples is prepared for transfer to the laboratory. The form will repeat the information on each of the sample labels and will serve as documentation of handling during shipment. The minimum information requirements of the COC form are listed in Table 1101.01-A. An example COC form is shown in Figure 1101.01-A. The completed COC must be reviewed by the Field Team Leader or Site Manager prior to sample shipment. The COC form will remain each sample shipping container at all times, and another copy will be retained by the member of the sampling team who originally relinquished the samples or in a project file.

SOP	1101.01				
GROUP	Sampling Handling		THE RESERVE		
SUB-GROUP	Sample Custody				
TITLE	Sample Custody in the	Field			
DATE	11/12/2003	FILE	1101-01.DOC	PAGE	3 of 4

TABLE 1101.01-A CHAIN OF CUSTODY FORM

INFORMATION	COMPLETED BY	DESCRIPTION		
COC	Laboratory	enter a unique number for each chain of custody form		
SHIP TO	Field Team	enter the laboratory name and address		
CARRIER	Field Team	enter the name of the transporter (e.g., FedEx) or handcarried		
AIRBILL	Field Team	enter the airbill number or transporter tracking number (if applicable)		
Project Name	Field Team	enter the project name		
Sampler Name	Field Team	enter the name of the person collecting the samples		
Sampler Signature	Field Team	signature of the person collecting the samples		
SEND RESULTS TO	Field Team	enter the name and address of the prime contractor		
FIELD Sample ID	Field Team	enter the unique identifying number given to the field sample (includes MS, MSD, iduplicate and field blanks)		
DATE	Field Team	enter the year and date the sample was collected in the format M/D (e.g., 6/3)		
TIME	Field Team	enter the time the sample was collected in 24 hour format (e.g., 0900)		
MATRIX	Field Team	enter the sample matrix (e.g., water, soil)		
PRESERVATIVE	Field Team	enter the preservative used (e.g., HNO3) or "none"		
FILTERED/ UNFILTERED	Field Team	enter "F" if the sample was filtered or "U" if the sample was not filtered		
CONTAINERS	Field Team	enter the number of containers associated with the sample		
MS/MSD	Field Team or Laboratory	enter "X" if the sample is designated for the MS/MSD		
ANALYSES REQUESTED	Field Team	enter the method name of the analysis requested (e.g., SW6010A)		
COMMENTS	Field Team	enter comments		
SAMPLE CONDITION UPON RECEIPT AT LABORATORY	Laboratory	enter any problems with the condition of any sample(s)		
COOLER TEMPERATURE	Laboratory	enter the internal temperature of the cooler, in degrees C, upon opening		
SPECIAL INSTRUCTIONS/COMME NTS	Laboratory	enter any special instructions or comments		
RELEASED BY (SIG)	Field Team and Laboratory	enter the signature of the person releasing custody of the samples		
COMPANY NAME	Field Team and Laboratory	enter the company name employing the person releasing/receiving custody		
RECEIVED BY (SIG)	Field Team and Laboratory	enter the signature of the person receiving custody of the samples		
DATE	Field Team and Laboratory	enter the date in the format M/D/YY (e.g., 6/3/96) when the samples were released/received		
TIME	Field Team and Laboratory	enter the date in 24 hour format (e.g., 0900) when the samples were released/received		

SOP	1101.01				
GROUP	Sampling Handling				
SUB-GROUP	Sample Custody				
TITLE	Sample Custody in the	Field			
DATE	11/12/2003	FILE	1101-01.DOC	PAGE	4 of 4

CHAIN OF CUSTODY FORM (SEE ATTACHMENT ON FOLLOWING PAGE)

SOP	1101.02				
GROUP	Sampling Procedures				
SUB-GROUP	Sample Handling	THE REAL PROPERTY.			1.5
TITLE	Sample Custody - Lab				
DATE	11/12/2003	FILE	1101-02.DOC	PAGE	l of 1

The following Standard Operating Procedure (SOP) presents the minimum requirements for sample handling and chain of custody (COC) procedures at an analytical laboratory.

PROCEDURE

Once the samples reach the laboratory, the person relinquishing the samples must sign the COC as well as the laboratory personnel receiving them. The date and time of sample receipt must be entered on the COC. The samples shall be checked against information on the COC form for anomalies. The condition, temperature, and appropriate preservation of samples shall be checked and documented on the COC form. The occurrence of any anomalies in the received samples and their resolution shall be documented in laboratory records. All sample information shall then be entered into a tracking system, and unique laboratory batch numbers and sample identifiers shall be assigned. This documentation will be considered as the sample receipt log. A copy of this information shall be reviewed by the laboratory manager for accuracy.

Subcontracted analyses and exchange of samples shall be documented with the original COC form. Procedures ensuring internal laboratory COC shall also be implemented and documented by the laboratory. Specific instructions concerning the analysis specified for each sample shall be communicated to the analysts. Analytical batches shall be created, and laboratory QC samples shall be introduced into each batch.

While in the laboratory, samples shall be stored in limited-access, temperature-controlled areas. Refrigerators, coolers, and freezers shall be monitored for temperature seven days a week. Acceptance criteria for the temperatures of the refrigerators and coolers is $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Acceptance criteria for the temperature of the freezers shall be less than 0°C . All of the cold storage areas shall be monitored by thermometers that have been calibrated with a NIST-traceable thermometer. As indicated by the findings of the calibration, correction factors shall be applied to each thermometer. Records that include acceptance criteria shall be maintained. Samples for volatile organics determination shall be stored separately from other samples, standards, and sample extracts. Samples shall be stored after analysis until disposed of IAW applicable local, state, and federal regulations. Disposal records shall be maintained by the laboratory.

The laboratory must maintain SOPs describing sample control and custody.

SOP	1102.01				
GROUP	Sample Handling				
SUB-GROUP	Sample Shipping				
TITLE	Sample Shipping				
DATE	11/12/2003	FILE	1102-01.DOC	PAGE	1 of 1

The following Standard Operating Procedure (SOP) presents the procedures for sample shipping that will be implemented during field work involving sampling activities.

TERMS

COC - Chain-of-Custody

PROCEDURE

Prior to shipping or transferring custody of samples, they will be packed according to D.O.T. requirements with sufficient ice to maintain an internal temperature of $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ during transport to the laboratory. Samples relinquished to the participating laboratories will be subject to the following procedures for transfer of custody and shipment:

- 1. Samples will be accompanied by a COC record. When transferring possession of samples, the individuals relinquishing and receiving the samples will sign, date, and note the time of the sample transfer on the record. If sent by common carrier, a bill of lading or airbill should be used. Bill of lading and airbill receipts will be retained in the project file as part of the permanent documentation of sample shipping and transfer. This custody record documents transfer of sample custody from the sampler to another person or to the laboratory. The designated laboratory will accept custody in the field upon sample pick-up or at the laboratory if the samples are delivered via field personnel or a courier service.
- 2. Samples will be properly packed in approved shipping containers for laboratory pick-up by the appropriate laboratory for analysis, with separate, signed custody records enclosed in each sample box or cooler. Sample shipping containers will be padlocked or custody-sealed for transfer to the laboratory. The preferred procedure includes use of a custody seal wrapped across filament tape that is wrapped around the package at least twice. The custody seal will then be folded over and stuck to itself so that the only access to the package is by cutting the filament tape or breaking the seal to unwrap the tape. The seal will then be signed. The designated laboratory will accept custody of the samples upon receipt.
- Whenever samples are split with state representatives or other parties, the COC record will be marked to indicate with whom the samples were split.
- The field sampler will call the designated laboratory to inform them of sample shipment and verify sample receipt as necessary.

SOP	1501.01							
GROUP	Field Documentation							
SUB-GROUP		ELLE						
TITLE	Field Logbook							
DATE	11/12/2003	FILE	1501-01.DOC	PAGE	1 of 3			

The following Standard Operating Procedure (SOP) presents the procedures for documenting activities observed or completed in the field in a field logbook. The documentation should represent all activities of WESTON personnel and entities under WESTON's supervision.

PROCEDURE

Field logbooks will be used and maintained during field activities to document pertinent information observed or completed by WESTON personnel or entities that WESTON is responsible for providing oversight. Field logbooks are legal documents that form the basis for later written reports and may serve as evidence in legal proceedings. The Site Manager or Field Team Leader will review field log entries daily and initial each page of entries. Field logbooks will be maintained by the Site Manager or Field Team Leader during field activities and transferred to the project files for a record of activities at the conclusion of the project. General logbook entry procedures are listed below.

- Logbooks must be permanently bound with all pages numbered to the end of the book.
 Entries should begin on page 1.
- · Only use blue or black ink (waterproof) for logbook entries.
- Sign entries at the end of the day, or before someone else writes in the logbook.
- If a complete page is not used, draw a line diagonally across the blank portion of the page and
 initial and date the bottom line.
- . If a line on the page is not completely filled, draw a horizontal line through the blank portion.
- · Ensure that the logbook clearly shows the sequence of the day's events.
- Do not write in the margins or between written lines, and do not leave blank pages to fill in
- If an error is made, make corrections by drawing a single line through the error and initialing
 it.
- Maintain control of the logbook and keep in a secure location.

Field logbooks will contain, at a minimum, the following information, if applicable:

General Information

- Name, location of site, and work order number
- · Name of the Site Manager or Field Team Leader

SOP	1501.01				
GROUP	Field Documentation				
SUB-GROUP			-4.19		
TITLE	Field Logbook				
DATE	11/12/2003	FILE	1501-01.DOC	PAGE	2 of 3

- Names and responsibilities of all field team members using the logbook (or involved with activities for which entries are being made)
- Weather conditions
- Field observations
- · Names of any site visitors including entities that they represent

Sample Collection Activities

- · Date(s) and times of the sample collection or event.
- · Number and types of collected samples.
- Sample location with an emphasis on any changes to documentation in governing documents (i.e., Work Plan, QAPP). This may include measurements from reference points or sketches of sample locations with respect to local features.
- Sample identification numbers, including any applicable cross-references to split samples or samples collected by another entity.
- A description of sampling methodology, or reference to any governing document (i.e. Work Plan, QAPP).
- · Summary of equipment preparation and decontamination procedures.
- Sample description including depth, color, texture, moisture content, and evidence of waste material or staining.
- Air monitoring (field screening) results.
- · Types of laboratory analyses requested.

Site Health and Safety Activities

- All safety, accident, and/or incident reports.
- · Real-time personnel air monitoring results, if applicable, or if not documented in the HASP.
- · Heat/cold stress monitoring data, if applicable.
- Reasons for upgrades or downgrades in personal protective equipment.
- Health and safety inspections, checklists (drilling safety guide), meetings/briefings.

SOP	1501.01				
GROUP	Field Documentation				
SUB-GROUP					
TITLE	Field Logbook				
DATE	11/12/2003	FILE	1501-01.DOC	PAGE	3 of 3

· Calibration records for field instruments.

Oversight Activities

- · Progress and activities performed by contractors including operating times.
- Deviations of contractor activities with respect to project governing documents (i.e., specifications).
- Contractor sampling results and disposition of contingent soil materials/stockpiles.
- Excavation specifications and locations of contractor confirmation samples.
- · General site housekeeping and safety issues by site contractors.

SOP	1502.01				
GROUP	Field Documentation				200
SUB-GROUP					
TITLE	Photograph Logs				100
DATE	11/12/2003	FILE	1502-01.DOC	PAGE	lofl

The following Standard Operating Procedure (SOP) presents the requirements for collecting information related to photodocumentation of site activities.

PROCEDURE

- Uniquely number each roll of film obtained for use.
- · Record the following information for each negative exposed:
 - 1. Date and Time
 - 2. Photographer Name
 - 3. Witness Name
 - 4. Orientation (Landscape, Portrait, or Panaoramic)
 - Description (including activity being performed, specific equipment of interest, sample location(s), compass direction photographer is facing)
- · Record "NA" for the negatives not used if the roll is not completely used prior to development.
- Record unique roll number on receipt when film is submitted for development.
- · Verify descriptions on log with negative numbers when photographs are received from processing.

FORMS

Blank Photograph Logs can be printed from WESTON On-Line from the Records Management Application. Selecting the Reports/Project Planning/Blank Photo Logs menu option will generate a project specific log with 36 entries.

SOP	1502.02							
GROUP	Field Documentation	Field Documentation						
SUB-GROUP			Maria di					
TITLE	Photograph Manageme	ent and Reporting						
DATE	11/12/2003	FILE	1502-02.DOC	PAGE	l of l			

The following Standard Operating Procedure (SOP) presents the requirements for managing and reporting information related to photodocumentation of site activities.

PROCEDURE

Enter the Photograph Log information specified in SOP 1502.01 into WESTON On-Line Records Management Application. The data entry screen can be accessed by selecting the Data/Photograph Log menu option.

REPORTS

Complete Photograph Logs can be printed from WESTON On-Line from the Records Management Application. Selecting the Reports/Summary Tables/Photographs/Logs menu option will generate a specific log for a selected roll of film.

Photograph Templates can be printed from WESTON On-Line from the Records Management Application. Selecting the Reports/Summary Tables/Photographs/Templates menu option will generate templates for mounting the photographs for a selected roll of film.

APPENDIX C

NIOSH Methods 1022 and 7903

(74 mm Hg; 9.8% v/v) @ 25 °C;

explosive range 11 to 41% v/v in air

CCI₂=CHCI MW: 131.39 CAS: 79-01-6 RTECS: KX4550000

METHOD: 1022, Issue 2 EVALUATION: PARTIAL Issue 1: 15 August 1987 Issue 2: 15 August 1994

suspect carcinogen; Group 1 Pesticide

ACGIH: 50 ppm; STEL 200 ppm; suspect carcinogen

 $(1 \text{ ppm} = 5.37 \text{ mg/m}^3 @ \text{NTP})$

RANGE STUDIED:

SYNONYMS: trichloroethene; ethylene trichloride; triclene

SAMPLING MEASUREMENT

SAMPLER: SOLID SORBENT TUBE TECHNIQUE: GAS CHROMATOGRAPHY, FID

(coconut shell charcoal, 100 mg/50 mg)

ANALYTE: trichloroethylene

FLOW RATE: 0.01 to 0.2 L/min

DESORPTION: 1 mL CS₂; stand 30 min

VOL-MIN: 1 L @ 100 ppm

-MAX: 30 L

INJECTION VOLUME: 5 μL

max. oo e

SHIPMENT: routine TEMPERATURE-INJECTION: 225 °C -DETECTOR: 250 °C

SAMPLE -COLUMN: 70 °C STABILITY: not determined

BLANKS: 2 to 10 field blanks per set

COLUMN: 3 m x 3-mm OD stainless steel, packed

with 10% OV-101 on 100/200 mesh

Chromosorb WHP

ACCURACY CALIBRATION: standard solutions of trichloroethylene in

477 to 2025 mg/m³

(3.4-L samples) [1] RANGE: 0.5 to 10 mg per sample

BIAS: - 7.19% ESTIMATED LOD: 0.01 mg per sample [2]

 OVERALL PRECISION (\hat{S}_{rT}):
 0.082 [1]

 ACCURACY:
 \pm 19.78%

 PRECISION (\hat{S}_{r}):
 0.038 @ 1.6 to 6.4 mg per sample [1]

APPLICABILITY: The working range is 27 to 875 ppm (150 to 4700 mg/m ³) for a 3.4-L air sample. The method is applicable to STEL determinations. The method was used for samples containing 0.5 to 5 mg trichloroethylene from a tool-degreasing operation [2].

INTERFERENCES: None studied. Alternate columns which have been used are stainless steel, 6 m x 3 mm OD, packed with 10% SP-1000 on 80/100 mesh Supelcoport [2] and fused silica capillary, 60 m x 0.32 mm, coated with 0.25 μ m OV-351 [3].

OTHER METHODS: This combines and revises methods S336 [4] and P&CAM 127 [5]. The criteria document method is similar [6]. NIOSH Method 3701 uses a portable gas chromatograph for field readout.

REAGENTS:

- 1. Carbon disulfide (CS ₂), chromatographic quality.*
- Trichloroethylene (TCE), reagent grade.*
- 3. Nitrogen, purified.
- 4. Hydrogen, prepurified.
- 5. Air, filtered, compressed.
 - * See SPECIAL PRECAUTIONS.

EQUIPMENT:

- Sampler: glass tube, 7 cm long, 6-mm OD, 4-mm ID, flame-sealed ends with plastic caps, containing two sections of 20/40 mesh activated (600 °C) coconut shell charcoal (front = 100 mg; back = 50 mg) separated by a 2-mm urethane foam plug. A silylated glass wool plug precedes the front section and a 3-mm urethane foam plug follows the back section. Pressure drop across the tube at 1 L/min airflow must be less than 3.4 kPa. Tubes are commercially available.
- 2. Personal sampling pump, 0.01 to 0.2 L/min, with flexible connecting tubing.
- 3. Gas chromatograph, flame ionization detector, integrator, and column (see page 1022-1).
- 4. Vials, 2-mL, PTFE-lined septum caps.
- 5. Syringes, 10-μL, readable to 0.1 μL.
- 6. Volumetric flasks, 10-mL.
- 7. Pipet, TD, 1-mL.

SPECIAL PRECAUTIONS: Carbon disulfide is toxic and a serious fire and explosion hazard (flash point = -30 °C). Trichloroethylene is a suspect carcinogen and a narcotic [6,7,8]. Work with these substances only in a hood.

SAMPLING:

- 1. Calibrate each personal sampling pump with a representative sampler in line.
- Break the ends of the sampler immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
- 3. Sample at an accurately known flow rate between 0.01 and 0.2 L/min for a total sample size of 1 to 30 L.
- 4. Cap the samplers. Pack securely for shipment.

SAMPLE PREPARATION:

- 5. Place the front and back sorbent sections of the sampler tube in separate vials. Discard the glass wool and foam plugs.
- 6. Add 1.0 mL CS ₂ to each vial. Cap each vial. NOTE: A suitable internal standard, such as ethylbenzene [1], undecane [2], or octane [3] at 0.1% (v/v) may be added at this step.
- 7. Allow to stand 30 min with occasional agitation.

CALIBRATION AND QUALITY CONTROL:

- 8. Calibrate daily with at least six working standards.
 - a. Add known amounts of TCE to CS $_2$ in 10-mL volumetric flasks and dilute to the mark. Use serial dilutions as needed to obtain TCE concentrations in the range 0.01 to 10 mg/mL.
 - b. Analyze with samples and blanks (steps 11 and 12).
 - c. Prepare calibration graph (peak area vs. mg TCE).

- Determine desorption efficiency (DE) at least once for each lot of sorbent used for sampling in the range of interest. Prepare three tubes at each of five concentrations plus three media blanks.
 - a. Remove and discard back sorbent section of a media blank sampler.
 - b. Inject a known amount (2 to 20 μL) of TCE, or a standard solution thereof in CS ₂, directly onto front sorbent section with a microliter syringe.
 - c. Cap the tube. Allow to stand overnight.
 - d. Desorb (steps 5 through 7) and analyze with working standards (steps 11 and 12).
 - e. Prepare a graph of DE vs. mg TCE recovered.
- 10. Analyze three quality control blind spikes and three analyst spikes to ensure that the calibration graph and DE graph are in control.

MEASUREMENT:

- Set gas chromatograph according to manufacturer's recommendations and to conditions given on page 1022-1. Inject sample aliquot manually using solvent flush technique or with autosampler.
 - NOTE: If peak area is above the linear range of the working standards, dilute an aliquot of the desorbed liquid with CS ₂, reanalyze, and apply the appropriate dilution factor in calculations.
- 12. Measure peak area.

CALCULATIONS:

- 13. Determine the mass, mg (corrected for DE) of TCE found in the sample front (W $_{\rm f}$) and back (W $_{\rm b}$) sorbent sections and in the average media blank front (B $_{\rm f}$) and back (B $_{\rm b}$) sorbent sections. NOTE: If W $_{\rm b}$ > W $_{\rm f}$ /10, report breakthrough and possible sample loss.
- 14. Calculate concentration, C, of TCE in the air volume sampled, V (L):

$$C = \frac{(W_f + W_b - B_f - B_b) \cdot 10^3}{V}, mg/m^3.$$

EVALUATION OF METHOD:

Method S336 was issued on June 6, 1975 [4], and validated with generated atmospheres using a calibrated syringe drive [1]. Average recoveries were 92 to 94% (16 samples) in the range 477 to 2025 mg/m 3 for 3.4-L samples. Breakthrough volume of 18.5 L (effluent = 5% of test concentration) occurred after sampling for 99 min at 0.187 L/min from an atmosphere containing 2266 mg/m 3 trichloroethylene in dry air. Desorption efficiency for SKC Lot 105 activated coconut charcoal in the range 1.6 to 6.4 mg per sample averaged 96.4% with $\bar{S}_r = 0.7\%$ (18 samples). n-Octane was used as an internal standard in the chromatographic measurements. The semi-quartile ranges of desorption efficiencies in two rounds of the Proficiency Analytical Testing (PAT) program were 0.97 to 1.0 for charcoal tubes spiked with 0.6 to 1.1 mg trichloroethylene [9].

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(1) HF; (2) HCl; (3) H₃PO₄; MW: Table 1 CAS: Table 1 RTECS: Table 1

(4) HBr; (5) HNO₃; (6) H₂SO₄

METHOD: 7903, Issue 2 EVALUATION: FULL Issue 1: 15 February 1984

Issue 2: 15 August 1994

OSHA: Table 1 PROPERTIES: Table 1

NIOSH: Table 1
ACGIH: Table 1

SYNONYMS: (1) hydrofluoric acid; hydrogen fluoride

(2) hydrochloric acid; hydrogen chloride

(3) phosphoric acid; ortho-phosphoric acid; meta-phosphoric acid

(4) hydrobromic acid; hydrogen bromide

(5) nitric acid; aqua fortis

(6) sulfuric acid; oil of vitriol

	SAMPLING		MEASUREMENT	
SAMPLER:	SOLID SORBENT TUBE (washed silica gel, 400 mg/200 mg with	TECHNIQUE:	ION CHROMATOGRAPHY F ⁻ , Cl ⁻ , PO ₄ ³⁻ , Br ⁻ , NO ₃ ⁻ , SO ₄ ²⁻	
	glass fiber filter plug)	ANALYTE:		
FLOW RATE:	0.2 to 0.5 L/min	DESORPTION:	10 mL 1.7 m <u>M</u> NaHCO ₃ /1.8 m <u>M</u> Na ₂ CO ₃	
VOL-MIN:	3 L			
-MAX:	100 L	INJECTION LOOP VOLUME:	50 μL	
SHIPMENT:	routine		•	
SAMPLE		ELUENT:	1.7 m \underline{M} NaHCO ₃ /1.8 m \underline{M} Na ₂ CO ₃ ; 3 mL/min	
STABILITY:	stable at least 21 days @ 25 °C [1]	COLUMNS:		
BLANKS:	ANKS: 2 to 10 field blanks per set		HPIC-AS4A anion separator, HPIC-AG4A guard, anion micro membrane suppressor [2]	
		CONDUCTIVITY		
	ACCURACY	SETTING:	10 μS full scale	
RANGE STUDIE	see EVALUATION OF METHOD	RANGE:	see EVALUATION OF METHOD	
BIAS:	see EVALUATION OF METHOD	ESTIMATED LOD:	see EVALUATION OF METHOD	
AYERALL PRE	ECISION ($\hat{\mathbf{S}}_{rT}$): see EVALUATION OF	PRECISION (S _r):	see EVALUATION OF METHOD	
ACCURACY:	± 12 to ± 23%			

APPLICABILITY: The working range is ca. 0.01 to 5 mg/m³ for a 50-L air sample (see EVALUATION OF METHOD). This method measures the total concentration of six airborne anions. The corresponding acids may be collected on a single sam pler and determined simultaneously. Formic acid has been determined by this method [3].

INTERFERENCES: Particulate salts of all the acids will give a positive interference. Chlorine or hypochlorite ion interfere with chloride determination and bromine interferes with bromide. Silica gel will collect ca. 30% of the free Cl₂ and Br₂ in an atmosphere [4]. Acetate, formate and propionate have elution times similar to F⁻ and Cl⁻. If these anions are present, use a weak eluent (e.g., $5 \text{ mM} \text{ Na}_{7}\text{B}_{4}\text{O}_{7}$) for greater resolution.

OTHER METHODS: This is P&CAM 339 in a revised format [5]. Alternate methods are 7902 for fluoride and P&CAM 268 for sulfate [6].

REAGENTS:

- 1. NaHCO₃, reagent grade.
- 2. Na₂CO₃, reagent grade.
- 3. Distilled, deionized water, filtered through 0.45-µm membrane filter.
- Eluent: bicarbonate/carbonate buffer solution (1.7 mM NaHCO₃/1.8 mM Na₂CO₃). Dissolve 0.5712 g NaHCO₃ and 0.7631 g Na₂CO₃ in 4 L filtered deionized water.
- Calibration stock solutions, 1 mg/mL (as the anion). Dissolve salt in filtered deionized water.
 - a. Fluoride: 0.2210 g NaF/100 mL.
 - b. Chloride: 0.2103 g KCl/100 mL.
 - c. Phosphate: 0.1433 g KH ₂PO₄/100 mL.
 - d. Bromide: 0.1288 g NaBr/100 mL.
 - e. Nitrate: 0.1371 g NaNO 3/100 mL.
 - f. Sulfate: 0.1814 g K ₂SO₄/100 mL.
 - * See SPECIAL PRECAUTIONS.

EQUIPMENT:

- Sampler: glass tube, 11 cm x 7-mm OD, containing a 400-mg front section and 200-mg backup section of washed silica gel, flame-sealed ends with plastic caps. Front section is retained with a glass fiber filter plug. Urethane plugs separate and retain the backup section. Tubes are commercially available (Supelco ORBO 53 or equivalent) or may be prepared according to APPENDIX.
- 2. Personal sampling pump, 0.2 to 0.5 L/min, with flexible connecting tubing.
- Ion chromatograph, HPIC-AG4A anion separator and HPIC-AG4A anion micro membrane suppressor, conductivity detector, integrator and strip chart recorder.
- Waterbath: hotplate with beaker of boiling water.
- Centrifuge tubes, 15-mL, graduated, plastic, with caps.*
- 6. Syringes, 10-mL, polyethylene with luer tip.
- 7. Filters, luer tip, with membrane filter, 13-mm, 0.8-µm pore size.
- 8. Micropipettes, disposable tips.
- 9. Volumetric flasks, 50- and 100-mL.*
- 10. Laboratory timer.
- 11. Bottles, polyethylene, 100-mL.
- 12. Auto sampler vials (optional).
 - * Thoroughly clean glassware with mild detergent, rinse thoroughly with deionized water, to minimize anion blank values.

SPECIAL PRECAUTIONS: Acids, particularly HF, are extremely corrosive to skin, eyes, and mucous membranes. HF will attack glass. Plastic labware is recommended.

SAMPLING:

- Calibrate each personal sampling pump with a representative sampler in line.
- 2. Break ends of sampler immediately before sampling. Attach sampler to personal sampling pump with flexible tubing.
- 3. Sample at an accurately known flow rate between 0.2 and 0.5 L/min for a total sample size of 3 to 100 L.

NOTE: Do not exceed 0.3 L/min when sampling for HF.

SAMPLE PREPARATION:

- 4. Score sampler with a file in front of primary sorbent section.
- 5. Break sampler at score line. Transfer glass fiber filter plug and front sorbent section to a 15-mL graduated centrifuge tube.

NOTE: Particulate salts of the volatile acids (HCl, HB, HF, and HNO ₃), if present in the air sample, will collect on the glass fiber filter plug. To estimate the concentration these salts, analyze the plug separately from the front sorbent section.

- 6. Place backup sorbent section in separate centrifuge tube. Discard urethane plugs.
- 7. Add 6 to 8 mL eluent to each centrifuge tube. Heat in boiling waterbath for 10 min. NOTE: Eluent used for desorption should be from same batch as the eluent used in the chromatograph to avoid carbonate/bicarbonate peaks near F and Cl-.
- 8. Allow to cool, dilute to 10.0-mL volume with eluent.
- 9. Cap the centrifuge tube and shake vigorously.
- 10. Pour sample into 10-mL plastic syringe fitted with in-line filter.

CALIBRATION AND QUALITY CONTROL:

- 11. Calibrate daily with at least six working standards covering the range 0.001 to 0.3 mg of each anion per sample.
 - Add known aliquots of calibration stock solution to eluent in 50-mL volumetric flasks and dilute to the mark.
 - b. Store working standards in tightly-capped polyethylene bottles. Prepare fresh working standards weekly.
 - c. Analyze working standards together with samples and blanks (steps 12 through 14).
 - d. Prepare a calibration graph for each anion [peak height (mm or μ S) vs. concentration (μ g per sample)].

MEASUREMENT:

- 12. Set ion chromatograph to conditions given on page 7903-1, according to manufacturer's instructions.
- 13. Inject 50-µL sample aliquot. For manual operation, inject 2 to 3 mL of sample from filter/syringe to ensure complete rinse of sample loop.
 - NOTE: All samples, eluents and water flowing through the IC must be filtered to avoid plugging system valves or columns.
- 14. Measure peak height.

NOTE: If sample peak height exceeds linear calibration range, dilute with eluent, reanalyze and apply the appropriate dilution factor in calculations.

CALCULATIONS:

- 15. Determine the mass, μg, of anion found in the sample front (W _t) and back (W _b) sorbent sections, and in the average media blank front (B _t) and back (B _b) sorbent sections.
- 16. Calculate concentration, C, of acid in the air volume sampled, V (L):

$$C = \frac{(W_f + W_b - B_f - B_b) \cdot F}{V}, mg/m^3.$$

where: F (conversion factor from anion to acid) = 1.053 for HF; 1.028 for HCl; 1.032 for H $_3$ PO $_4$; 1.012 for HBr; 1.016 for HNO $_3$; and 1.021 for H $_2$ SO $_4$.

EVALUATION OF METHOD:

The method was evaluated for hydrochloric, hydrobromic, nitric, phosphoric and sulfuric acids by laboratory generation of mixed acids [1]. Data for the individual analytes are:

Acid	Range (mg/m³)	e Studied (µg/sample)	<u>(%)</u>	Measurement Precision (S ,)	Overall Precision _ <u>(Ŝ_{rT})</u>	Accuracy _(%)	Estimated LOD [2] (µg per sample)
HF [7]	0.35 - 6	0.5 - 200	0.7	0.053	0.116	±23.4	0.7
HCI [8]	0.14 - 14	0.5 - 200	0.3	0.025	0.059	±11.9	0.6
H ₃ PO ₄ [1]	0.5 - 2	3 - 100	-0.9	0.029	0.096	±19.7	2.0
HBr [1]	2 - 20	3 - 960	2.0	0.056	0.074	±16.5	0.9
HNO ₃ [1]	1 - 10	3 - 500	2.0	0.018	0.085	±18.7	0.7
H ₂ SO ₄ [1]	0.5 - 2	3 - 100	2.4	0.028	0.087	±19.4	0.9

The method was field-evaluated at two electroplating facilities using side-by-side silica gel tubes and bubblers. The method was evaluated for hydrofluoric acid in 1983 using the silica gel tubes and impingers [7]. Recovery based on impinger collection was 106% with $\,\hat{S}_{rT}$ of 0.116. The capacity of the silica gel sampler for HF was 820 μ g. This is equivalent to an 8-h sample at two to three times the OSHA PEL. Samples were stable for at least 21 days at 25 °C. Updated analytical columns have been used by NIOSH for analytical sequences [2].

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APPENDIX: SAMPLING TUBE PREPARATION

Silica gel cleaning procedure: Add 500 to 600 mL deionized water, slowly and with stirring, to ca. 200 mL volume of silica gel in 1-L beaker. When exothermal reaction has subsided, heat in boiling waterbath for ca. 30 min with occasional stirring. Decant and rinse four to five times with deionized water. Repeat cleaning procedure and dry overnight in 100 °C oven until free flowing. If blank of silica gel shows impurities upon analysis by ion chromatography, repeat cleaning procedure.

Silica gel tubes: Pack glass tubes, 7-mm OD, 4.8-mm ID, 11 cm long, with 400 mg of 20/40 mesh washed silica gel in front section and 200 mg backup section. Use urethane foam plugs between sorbent sections and at back end. Hold front section in place with 6-mm diameter, 1-mm thick glass fiber filter plug (Gelman 66088).

TABLE 1. GENERAL INFORMATION.

Acid			EXPOSURE	LIMITS			
and	CAS	VD 0 00 00	_		mg/m³ =	Physical	MP
BP <u>M.W.</u> (°C)	Sp. Gr. <u>RTECS</u> (liq.)	VP @ 20 °C OSHA kPa_(mm_Hg)	NIOSH_	ACGIH	<u>1 ppm @ NTP</u>	_State	_(°C)_
HF 19.5 (20.01)	7664-39-3 0.987 MW7875000	3 ppm >101 (>760)	3 ppm; 6 ppm STEL	C 3 ppm;	0.818	gas	-83.1
HCI -114.8 (36.46)	7647-01-0 -85.0 MW4025000	C 5 ppm 1.194	C 5 ppm >101 (760)	C 5 ppm	1.491	gas	
H ₃ PO ₄ 260 (97.99)	7664-38-2 1.7 TB6300000	1 mg/m ³ 0.0038 (0.03)	1 mg/m ³ ;* STEL 3 mg/m ³	1 mg/m ³ ; STEL 3 mg/m ³	(aerosol)	liquid	21.0
HBr -66.8 (80.92)	10035-10-6 2.16 MW3850000	3 ppm >101 (>760)	C 3 ppm	C 3 ppm	3.31	gas	-88.5
HNO ₃ -42.0 (63.01)	7697-37-2 83 QU5775000	2 ppm 1.50	2 ppm; 0.39 (2.9) STEL 4 ppm	2 ppm; STEL 4 ppm	2.58	liquid	
H ₂ SO ₄ 290 (98.08)	7664-93-9 1.84 W55600000	1 mg/m ³ <0.0001 (<0.001)	1 mg/m³*	1 mg/m ³ STEL 3 mg/m ³	(aerosol)	liquid	3.0

^{*}Group I Pesticide